

CLAIMS

1. Process for the preparation of an insertion compound of an alkali metal in which the following successive stages are carried out:
 - a) an organic complex of a transition metal or of a mixture of transition metals M in an oxidation state of greater than 2 is brought into contact with an alkali metal A in the ionic form and with an entity of formula $H_b(XO_4)$, where X is chosen from Si, S, Al, P, Ge, As or Mo and b has a value from 0 to 5, in a liquid medium in a closed chamber; the chamber is brought to a temperature T which makes possible the decomposition of the organic complex in the the said liquid medium;
 - b) the temperature and the pressure in the chamber are brought back to ambient temperature and atmospheric pressure and the insertion compound of an alkali metal of formula $AMXO_4$, in which M is in the +2 oxidation state, is recovered.
2. Process according to Claim 1, in which the metal M is in an oxidation state of 3 to 5, preferably in an oxidation state equal to 3.
3. Process according to either one of the preceding claims, in which M is chosen from transition metals, such as Mn, Fe, Ni, Co and their mixtures.
4. Process according to any one of the preceding claims, in which the alkali metal A is chosen from Li and Na.
5. Process according to any one of the preceding

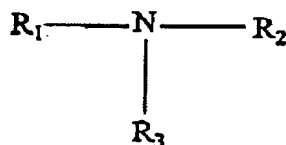
claims, in which the alkali metal A in the ionic form is in the form of an alkali metal salt $A_aH_b(XO_4)$ where a has a value from 1 to 5.

5 6. Process according to any one of the preceding claims, in which X is P.

7. Process according to any one of Claims 4 to 6, in which the alkali metal salt is Li_2HPO_4 .

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8. Process according to any one of the preceding claims, in which the organic complex comprises the metal M bonded to an organic ligand chosen from the compounds of formula:



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in which at least one from R_1 , R_2 and R_3 comprises at least one oxygen atom.

9. Process according to Claim 8, in which, in the organic ligand, R_1 , R_2 and R_3 are chosen independently from carboxy(1-4C) alkyl radicals, such as carboxymethyl and carboxyethyl.

10. Process according to Claim 9, in which the organic ligand is nitrilotriacetic acid $N(CH_2CO_2H)_3$ or ethylenedioxyethylenedinitriletetraacetic acid (EGTA).

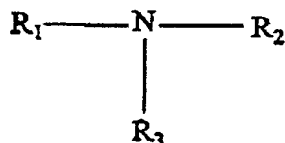
11. Process according to any one of the preceding claims, in which the liquid medium, in stage a), is chosen from water; organic solvents, such as liquid alkanes, for example dodecane, or tributyl

phosphate (TBP); and their mixtures.

12. Process according to any one of the preceding claims, in which, on conclusion of stage b), the compound is washed and then dried, optionally under vacuum.

13. Process according to any one of the preceding claims, in which the organic complex is prepared in a stage prior to stage a) by bringing a salt of the metal M, in the oxidation state greater than 2, into contact with an organic compound in a liquid medium.

14. Process according to Claim 13, in which the organic compound is chosen from the compounds of formula:



in which at least one from R_1 , R_2 and R_3 comprises at least one oxygen atom.

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15. Process according to Claim 14, in which, in the organic compound, R_1 , R_2 and R_3 are chosen independently from carboxy(1-4C)alkyl radicals, such as carboxymethyl and carboxyethyl.

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16. Process according to Claim 15, in which the organic compound is nitrilotriacetic acid $N(CH_2CO_2H)_3$ or ethylenedioxyethylenedinitrile-tetraacetic acid.

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17. Process according to any one of Claims 13 to 16,

in which the salt of the metal M is chosen from nitrates, sulphates, chlorides, acetates, citrates or carboxylates of the metal M.

- 5 18. Process according to any one of Claims 13 to 17, in which the liquid medium is chosen from water; organic solvents, such as liquid alkanes, for example dodecane, or tributyl phosphate (TBP); and their mixtures.
- 10 19. Insertion compound of an alkali metal of formula $AM(XO_4)$ where A is chosen from alkali metals, X is chosen from Si, S, Al, P, Ge, As and Mo, and M is in the +2 oxidation state, characterized in that it exhibits a content of metal M as at oxidation
- 15 state of greater than 2, for example of metal M(III), of less than 5% by weight, preferably of less than 1% by weight.
- 20 20. Compound according to Claim 19, which is present in the form of particles or grains.
- 25 21. Compound according to Claim 20, in which the particles have the shape of cylinders, cubes or polyhedra.
22. Compound according to either one of Claims 20 and 21, in which the particles exhibit a fully controlled, homogeneous, morphology.
- 30 23. Compound according to any one of Claims 20 to 22, in which the deviation from the mean value of the size of the particles is less than 20%, preferably less than 10%, more preferably less than 1%.

24. Electrode active material comprising one or more compounds according to any one of Claims 19 to 23 or prepared by the process according to any one of Claims 1 to 18, optionally in combination with one or more other active compounds, such as LiCoO_2 , LiNiO_2 , manganese oxides, in particular with the spinel structure $\text{Li}_{1+x}\text{Mn}_{2-x}\text{O}_4$ (with $0 \leq x \leq 0.33$), for example LiMn_2O_4 , compounds of the family isotypic with olivine, such as $\text{Li}_{1-x}\text{FePO}_4$, for example LiFePO_4 , compounds with the Nasicon structure and the insertion materials of lithium of the orthosilicate type.
25. Positive electrode comprising the active material according to Claim 24.
26. Battery comprising the electrode according to Claim 25.
27. Battery according to Claim 26, comprising a negative electrode based on $\text{Li}_4\text{Ti}_5\text{O}_{12}$.
28. Electrochromic device comprising the compound according to any one of Claims 19 to 23 or prepared by the process according to any one of Claims 1 to 18.